ESTCP
Cost and Performance Report

(CU-0130)

Applied Innovative Technologies for Characterization of Explosives-Contaminated DoD Building Foundations and Underlying Soils

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ACRONYMS AND ABBREVIATIONS

BAAP	Badger Army Ammunition Plant
CRREL	Cold Regions Research and Engineering Laboratory
DNT	dinitrotoluene
DoD	Department of Defense
ESTCP	Environmental Security Technology Certification Program
mg/kg	milligram(s) per kilogram
µg/g	microgram(s) per gram
NC	nitrocellulose
NG	nitroglycerine
nm	nanometer(s)
RDX	Royal Demolition Explosive
RP	Rocket Paste
RPD	relative percent difference
SASW	Spectral Analysis of Surface Waves
SVOC	semivolatile organic compound
TNT	Trinitrotoluene
USEPA	U.S. Environmental Protection Agency
WDNR	Wisconsin Department of Natural Resources

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1.0 EXECUTIVE SUMMARY

1.1 BACKGROUND INFORMATION

Badger Army Ammunition Plant (BAAP) is included in the first group of 13 inactive Army ammunition plants with transitioning missions. Installation Management Agency currently manages these plants and is in varying stages of transferring the properties out of Department of Defense (DoD) control. To accomplish this, many buildings used in the production, loading, handling, and storage of explosives must be demolished or characterized and decontaminated to a level protective of human health and the environment. BAAP alone has more than 1,400 buildings on the installation that will have to be addressed. Compounds associated with the buildings include nitrocellulose (NC), nitroglycerine (NG), dinitrotoluene (DNT), and common compounds such as asbestos-containing material, solvents, and metals.

There are no full-scale technologies for nondestructive in situ characterization of hard to reach surfaces (e.g., under floors) in explosive-contaminated buildings. With adequate characterization, many buildings could be safely left in place, avoiding substantial costs and speeding up the transfer of the properties out of DoD control. This Environmental Security Technology Certification Program (ESTCP) demonstration evaluated a variety of methods for characterizing the foundations, adjacent areas, and underlying soils without having to remove the buildings and foundations first. The intent of this demonstration was to show that the evaluated methods could be applied to many of the buildings at BAAP and at similar sites throughout the United States.

As part of this demonstration, field test methods including Raman spectroscopy, Expray® colorimetric indicator, and the Cold Regions Research and Engineering Laboratory (CRREL) Royal Demolition Explosive (RDX) colorimetric field screening method were evaluated at five buildings in the Rocket Paste (RP) area at BAAP to determine the presence and/or concentration of NC or NG in soil samples and concrete slabs. Results from these field measurements were compared to laboratory analyses of NC and NG in the same materials to evaluate the reliability of the field screening and analytical methods for identifying and quantifying NC and NG in building foundations and soil. Raman spectroscopy was also evaluated for identifying the presence of other organic compounds used in the manufacturing processes within the study area. The demonstrations were conducted April through May 2002 and August 2002.

1.2 OBJECTIVES OF THE DEMONSTRATION

The objectives of the demonstration were to evaluate the reliability of field instrumentation and analytical techniques for identifying and measuring NC and NG on building foundations and in underlying soils and to compare the field results to reference laboratory analytical methods. The demonstration was mainly conducted on NC and NG but could be used for other explosive compounds, such as RDX. Potential NC and NG contamination in soils, on building foundation surfaces, and in cracks and flaws in concrete floors were investigated.

The goals of the demonstration were not achieved to the degree envisioned. Because of the small number of actual positive results and the problems associated with the NC analyses, rigorous statistical comparison between the analytical methods was not possible.

1.3 REGULATORY DRIVERS

Safety concerns related to the explosive nature of NC and NG provided the driver for this investigation. OSC Regulation 385-1 addresses explosive safety at all Army industrial facilities and requires buildings to be classified and remediated, if necessary, prior to transfer. The regulation provides guidance on detecting explosive contamination, determining the contamination status, recommending decontamination methods, and marking contaminated items. Buildings may not be released to the public until they do not pose an explosive safety hazard and are safe for welding, drilling, sawing, and sale to the general public. OSC Regulation 385-1 defines the amount of explosives required to create an explosive safety hazard as dependent on the properties of the explosive, the concentration or distribution of the contaminant on the surface, and the amount of confinement in the potential incident. In *Testing to Determine Relationship Between Explosive Contaminated Sludge Components and Reactivity*[1], Army considers soils containing concentrations of primary explosives, such as NG, in excess of 4 percent to be an explosive hazard. In *Analysis, Preliminary Determination and Draft Plan on the Explosive Decontamination and Demolition at* BAAP[2] regarding open burning of buildings at BAAP, the Wisconsin Department of Natural Resources (WDNR) also cited OSC Regulation 385-1 with regard to explosive safety at BAAP. No numerical limits pertaining to concentrations of NC or NG were included with this citation.

There are no state or federal numerical environmental standards for NC and NG cleanup within soils and building materials. NG is both a federal and Wisconsin listed waste (P081) and is considered a possible carcinogen by the U.S. Environmental Protection Agency (USEPA). There is also no DoD standard for NC and NG residual contamination.

1.4 DEMONSTRATION RESULTS

Several problems were encountered during the demonstration, including an apparent lack of significant quantities of energetic compounds[1] at the sampling locations and buildings, access and sampling issues for the concrete cutting equipment, and failure of the laboratory reference method for NC. These problems made it difficult to assess whether the CRREL RDX Method, Expray®, and Raman spectroscopy could be implemented at other DoD sites.

The Raman equipment requires concentrations of energetic material of at least 1% (10,000 milligrams per kilogram [mg/kg]) in soils for detection. Since none of the soils encountered during the study contained concentrations close to that range, the study was unable to quantify levels of explosives that might be reliably detected using Raman spectroscopy. Use of Raman

[1] Throughout this document, quantities of energetic materials are discussed in light of the explosive safety concerns that provided the driver for the demonstration. OSC Regulation 385-1 and *Testing to Determine Relationship Between Explosive Contaminated Sludge Components and* Reactivity[1] provide guidance on amounts of energetic materials (4% or 40,000 mg/kg). Although NC and/or NG may have been present at very low levels at the demonstration sample locations, no samples indicated NC or NG at levels approaching concentrations that would cause a safety concern.

spectroscopy for investigations not related to explosive safety (i.e., regulatory compliance) may be limited, especially if regulatory compliance limits are below the 1% limit.

Expray® analysis did detect the presence of explosive compounds in various areas at BAAP. It appears that there are potentially many more false positive results with Expray® than with other methods, but the false negative rate for the Expray® analysis was 0% when compared with the laboratory reference methods (assuming that low-level, qualified results show lack of significant quantities of explosives). In certain situations, Expray® may still be used to indicate lack of explosive compounds, given its low rate of false negatives. Further validation is needed to ensure that methods completely perform within performance metrics with valid reference method confirmation.

Based on the limited data generated in the spiked sample study, it appears that the CRREL RDX method can reliably detect and quantify NG in soils. It also appears that the method can detect NC in soils, but the method cannot adequately measure NC concentrations. The CRREL RDX method as it currently stands cannot be used as a field-screening tool for surveying explosive residues (particularly NC) in DoD buildings.

Overall, based on the results of the demonstration, the technologies evaluated require additional validation and cannot be recommended for full-scale implementation at DoD sites.

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2.0 TECHNOLOGY DESCRIPTION

Three distinct technologies for identifying and quantifying NC and NG were evaluated against laboratory analysis. These included:

- Raman spectroscopy,
- Expray® colorimetric indicator, and
- CRREL RDX colorimetric field screening method.

Each of these technologies is addressed in the following sections.

2.1 RAMAN SPECTROSCOPY

2.1.1 Description — Theory, Functionality, and Operation

Raman spectroscopy technology has been developed and proven for detecting and identifying chemicals through inelastic scattering of incident light. The basis for this technology derives from the observation that a small fraction of the light falling on a material will be absorbed by exciting the chemical bonds in the molecule, resulting in molecular vibration. When the molecule returns to its ground state, this excitation energy is released as a photon. This photon will have an energy different from the incident light and the characteristic of the chemical bond that was previously excited. Each chemical will therefore have a characteristic Raman spectrum.

Recent advances in laser technology and signal processing have resulted in miniaturization of Raman spectroscopy devices. The device that was evaluated in the course of this project was approximately 1 inch in diameter. The probe contained a laser for illuminating the sample and was connected to the signal processing unit by way of a fiber optic cable so the probe could be deployed into remote locations.

Raman spectroscopy is not significantly limited by sample size, particularly in the case of solids. The intensity of the Raman scattered light is essentially constant for any size particle larger than the wavelength of the incident illumination. The Raman spectroscopy unit evaluated in this study used an illumination wavelength of 632 nanometers (nm), making it possible to detect and identify submicrogram particles of NC or NG.

The Raman spectroscopy probe can be used under adverse circumstances, including wet conditions and limited access, and it is capable of resolving and identifying particles in a complex matrix such as soil or concrete. The selected Raman spectroscopy probe is also capable of detecting NC or NG on building or equipment surfaces, and it can be pushed into soil to detect NC and NG particles below the soil surface.

Raman spectroscopy has been used to identify organic chemicals since its discovery in the late 1920s. Recent developments over the last decade in laser technology, signal processing, and miniaturization has greatly enhanced the capability of the technique. Rugged field deployable Raman spectroscopy units have been available for use since approximately 1998. Potential applications for the technology include a wide range of measurement techniques for both

qualitative and quantitative measurements of organic and inorganic chemicals. Liquids, solids, gases, and vapors can be analyzed. Measurements can be made at room temperature as well as at very low or high temperatures.

2.1.2 Strengths, Advantages, and Weaknesses

The significant advantages of this technology are that positive identification of the target compounds NC and NG can be made in the field. This provides an advantage over current laboratory techniques, which require sampling, packaging, shipping, and analysis of the sample. Determinations can be made in situ without disturbing the material. The system can be deployed to remote or inaccessible locations including pipes, cracks, and other irregularities that may serve as collection points. This provides an advantage over conventional investigation, which would require removal of portions of the structure being analyzed to access the sampling locations. The technology is extremely sensitive, capable of detecting submicrogram particles of NC or NG. However, this sensitivity is limited by the heterogeneity of the soil. Typically, Raman spectroscopy requires concentrations of energetic materials of 10,000 mg/kg (>1%) for detection. The high detection limit of this technology may limit its application on low concentration samples (<1%).

The principal drawback of the technology is that while it provides positive identification of NC or NG, it does not provide quantitative information. The system may not be capable of distinguishing trivial quantities of NC and NG from quantities requiring remediation. The probe, while small, cannot access locations smaller than a 1-inch width or certain locations where the geometry of the location prohibits a probe.

2.1.3 Factors Influencing Cost and Performance

The principal factor affecting cost and performance is the reliability of the instrument and the system as a whole. The Raman spectroscopy system generally performs reliably as long as the system components (i.e., computer, probe, etc.) are all functioning in unison. This is generally a matter of having the equipment supplier properly integrate the equipment before shipping it to the field. Performance is not affected by distance between the probe and the signal processing unit. Typically, five to ten Raman analyses can be performed readily in an hour under normal field circumstances.

2.2 EXPRAY® COLORIMETRIC INDICATOR

2.2.1 Description — Theory, Functionality, and Operation

The Expray® system consists of a set of three aerosol sprays. The sprays are used in a fixed sequence to identify a variety of explosive compounds, including NC and NG. Explosive compound identification is performed colorimetrically.

Expray® is applied to a filter paper that has been previously wiped over a suspect surface. The wipe is then sprayed with the first spray. A color change indicates the presence of trinitrotoluene (TNT) (dark brown/violet), DNT (blue-green), or trinitrobenzene, picric acid, or other Group-A explosives (orange). If the first spray does not react, the second spray is applied. A pink color

indicates the presence of Group-B explosives, including NC and NG. Following this, the third spray is applied, which will indicate the presence of inorganic nitrates.

Expray® is a detect/nondetect method. Performance evaluations conducted by Sandia National Laboratories[3], [4] show a detection level for TNT at +200 nanograms total sample, although the manufacturer claims a detection level less than this.

Expray® has been used for several years for screening persons, baggage, and other items at transportation facilities. It has also been used in forensic applications to identify the presence of explosives. A modification of this method has been developed for testing for the presence of explosives in soil.

2.2.2 Strengths, Advantages, and Weaknesses

Expray® is a demonstrated technology for identifying a wide variety of explosive compounds in the field. This technology gives a qualitative indication of whether explosive compounds exist at the testing site and provides information on what type of explosive compounds have been detected. This provides an advantage over conventional analysis, which requires sampling, packaging, shipping, and analysis of the sample. Expray® is rapid and has a low rate of false negatives.

Disadvantages of the technology are that the method is not quantitative and the identification is not specific for either NC or NG. Also, some interferences have been observed in the field, causing development of other colors that may mask positives or be misinterpreted as explosives.

2.2.3 Factors Influencing Cost and Performance

No factors have been identified that affect cost and performance. Typically, 30 Expray® analyses can readily be performed in an hour under normal field circumstances.

2.3 CRREL RDX METHOD

2.3.1 Description — Theory, Functionality, and Operation

The CRREL RDX method is a colorimetric quantitative field portable analytical method for identifying and quantifying RDX and certain other explosives, including pentaerythritol tetranitrate, high melting explosive, NC, NG, and tetryl. The method involves extracting the soil (or other solid material) with acetone to remove the explosive compounds. The extract is filtered, acidified, and treated with zinc dust. Treatment with acid and zinc liberates the nitro groups from the compound as nitrite ions. Nitrite is then quantified using Hach Chemical Company's proprietary NitroVer 3® reagent, which reacts with nitrite to form a pink color whose intensity is proportional to the concentration of nitrite. The absorbance of the treated extract is measured at a wavelength of 507 nm.

This method was developed by CRREL, a branch of the U.S. Army Corps of Engineers Research and Development Center. It was originally published in *Development of a Field Screening Method for RDX in* Soil[5] in 1991. The authors and others have used the method extensively

for evaluating soils contaminated with RDX. The method is also in the process of adoption by the USEPA as SW-846 Method 8510.

Potential applications for the technology include quantitative field analysis of NC and NG at locations where RDX is not present, as is the case at BAAP and similar sites.

2.3.2 Strengths, Advantages, and Weaknesses

The advantage of the method lies in its speed, simplicity, and low cost. The method can be used with minimal training and produces a numerical quantitative value for the explosive compounds in the soil based on the quantity of nitrite present.

The method is not specific for NC or NG and was developed and used for RDX quantitation. Since NC and NG are the only explosives present at the demonstration site, this does not constitute a technical problem.

The method cannot distinguish between NC and NG, since the extract treatment destroys both of the parent compounds, liberating the nitro groups from both. NC is not a discrete molecule of constant composition. Consequently, the method is not strictly quantitative for NC. Nitrite measured in the extract must be converted to an arbitrary average value NC equivalent that may differ in quantity from the NC present in the sample.

2.3.3 Factors Influencing Cost and Performance

No factors have been identified that affect cost and performance. Typically, five soil samples can be analyzed in an hour under normal field circumstances using the CRREL RDX method.

3.0 DEMONSTRATION DESIGN

3.1 PERFORMANCE OBJECTIVES

Structures, facilities, and property that have been or may have been contaminated by propellants and explosives must be cleaned up with regard to the explosive material before they can be sold, transferred, or otherwise disposed of in a manner that may result in public exposure. The Army considers soils containing concentrations of primary explosives, such as NG, in excess of 4% to be an explosive safety hazard. Propellants, such as NC, with moisture content below 30% can burn when exposed to an ignition source such as a spark.

The performance objective for this study was to quantify the levels of propellants and/or explosives that may be reliably detected using specific field test methods. If this quantitation level was sufficiently low and reliable, these field tests might then be used to support decisions relating to required remediation or to release buildings for unrestricted public use or access.

The goals of the demonstration were not achieved to the degree envisioned. Because of the small number of actual positive results and the problems associated with the NC analyses, rigorous statistical comparison between the analytical methods was not possible. Performance objectives are summarized in Table 1.

Table 1. Performance Objectives.

Type of Performance Objective	Primary Performance Criteria	Expected Performance
Qualitative	1. Identify areas that require decontamination	Detection confirmed by laboratory analysis
	2. Identify areas that do not require decontamination	Absence confirmed by laboratory analysis
	3. Determine frequency of false negatives/positives for Expray® and Raman spectroscopy	Not more than 5% based on laboratory analysis
	4. Ease of use	Operator acceptance
Quantitative	1. Areal concentration	1 µg/100 cm^2
	2. Mass concentration	1 µg/g
	3. Agreement with reference laboratory methods for CRREL RDX method, using statistical correlation methods	+/- 50% and/or correlation coefficient >0.95
	4. Method detection limit	To be determined

3.2 SELECTION OF TEST SITES

The test sites for performing the technology evaluation were selected using the following criteria.

- Structures were used directly in the manufacture of explosives, specifically NC and/or NG.
- Test sites were representative of a variety of potentially contaminated sites.

- Physical condition of structures exhibited some deterioration and irregularities, particularly in the foundations.

- For purposes of evaluating the overall reliability of the test methods, selected test sites had a strong potential for containing a wide range of NC and NG concentrations, including a sufficient number of uncontaminated locations.

- Test sites provided ample locations that likely served as specific accumulators of NC or NG residue.

The buildings previously used for the production of RP were selected as fulfilling all these criteria. The activities that occurred in the buildings generated large quantities of dust. The dust has been found in the wooden frame parts of these buildings, and it was anticipated that the dust would be found in or beneath the cracks in the concrete floors. The floors of all five buildings were regularly washed down with water and/or neutralizing solutions, which may also have carried RP compounds into the cracks or may have spilled into the soils under the gutters leading from the buildings.

3.3 SITE/FACILITY CHARACTERISTICS

The RP area was constructed between 1944 and 1945 for the manufacture of rocket propellants. RP is used to manufacture a double-based plasticized nitrocellulose propellant used in rockets. The final propellant contains NC, NG, plasticizers, and burn rate modifiers that are added during various mixing stages of the process. The rocket propellant manufacturing process at BAAP was performed in three major processing areas: the Paste Area, Rolls and Press Area, and Finishing Area. These areas contain numerous buildings for blending, drying, pressing, and milling propellant. Visible RP was removed from the buildings and burned at the Propellant Burning Ground after BAAP went on standby status. However, potential accumulation of propellant within, around, and under the buildings' structural foundations has not been addressed. Project building locations are shown in Figure 1.

3.4 PHYSICAL SET-UP AND OPERATION

The dissimilarity of the buildings required that each building be characterized differently. However, the general process of the characterization was the same in each case. It consisted of five basic activities, including a records research, field visual inspection, the characterization of features in the concrete using Spectral Analysis of Surface Waves (SASW), classification of test locations, and sampling and analysis.

The process flow for project activities is summarized diagrammatically in Figure 1. It is described in detail in the Demonstration Plan[6]. The timeline for the demonstration is provided in Table 2.

Figure 1. Project Building Locations.

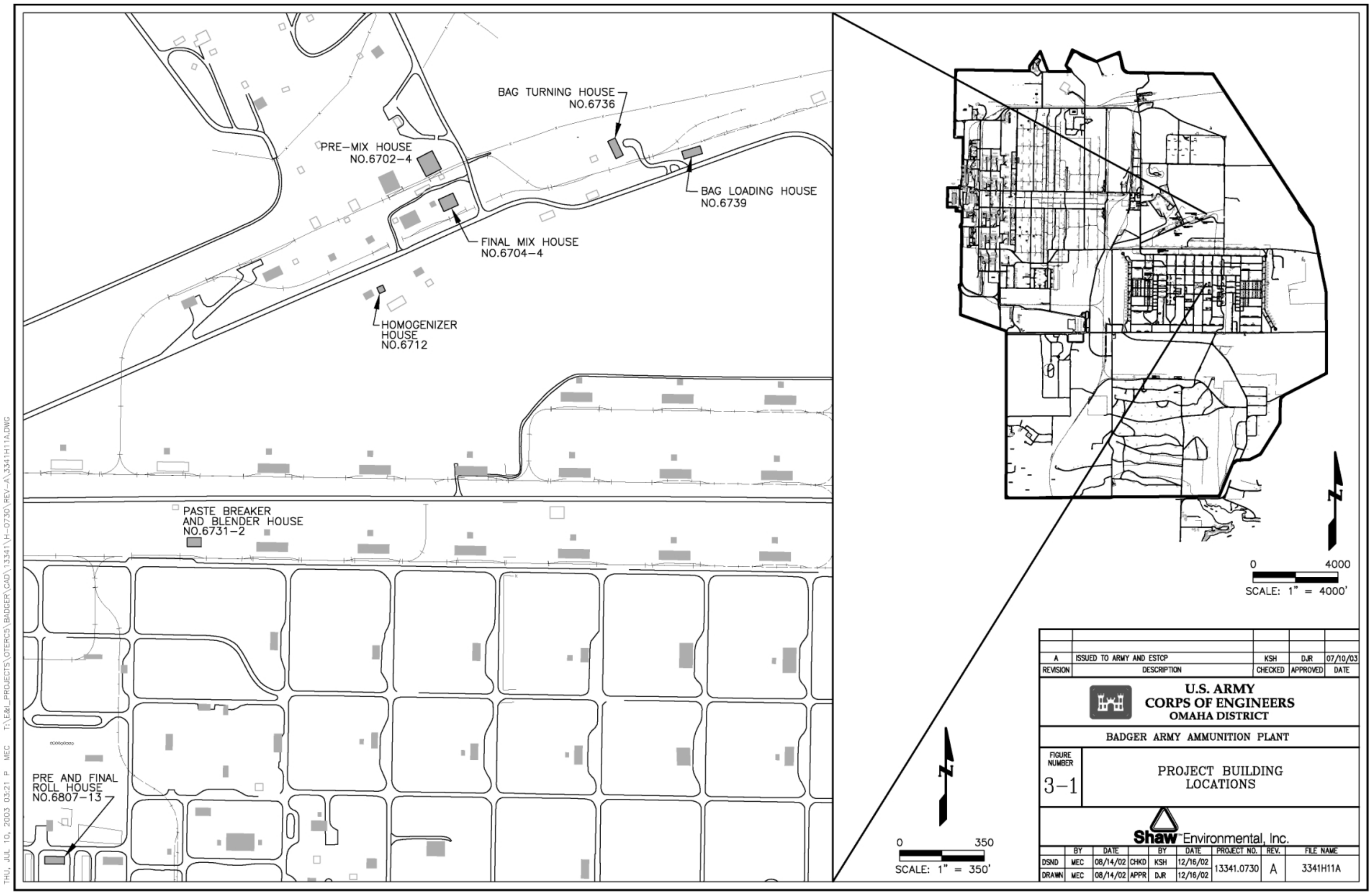

Table 2. ESTCP Demonstration Field Timeline.

Activity	Start Date	End Date
Mobilization	4/28/02	4/29/02
Building inspection/grid layout	4/29/02	5/2/02
SASW testing	4/30/02	5/3/02
Expray® wipe sampling	5/3/02	5/3/02
Surface Raman testing	5/6/02	5/7/02
Concrete coring	5/8/02	5/11/02
Subsurface Raman testing	5/9/02	5/11/02
Soil sampling	5/9/02	5/11/02
SW-846 8510 field testing	5/9/02	5/13/02
Demobilization	5/13/02	5/13/02
Additional soil sampling	8/27/02	8/28/02

A number of activities took place before sampling. A review of available records was performed to identify potential sampling locations in the building. Following this, each building's foundation, floor, and associated structures (e.g., drain troughs and external catch tanks) were visually inspected. All locations that presented potential pathways for solids or liquids to get into or beneath the floor or foundation were noted on the field drawing. Each crack in the concrete floor that was identified on the inspection log was examined along its length at 2-foot intervals using the SASW equipment to determine the extent of the fissure. The SASW testing was used to determine if the fault extended completely through the slab, or if it terminated within the thickness of the concrete. The result of the inspections described above was a comprehensive list of locations that required additional investigation. Each identified feature requiring additional characterization was classified as a narrow nonpenetrating crack, a wide nonpenetrating crack, or a penetrating crack. The type of feature determined which investigative method was applied to it (refer to Figure 2).

3.5 SAMPLING/MONITORING

Sampling procedures used during the field demonstration are described in the following paragraphs. For more detail about field sampling procedures, please refer to the *Demonstration* Plan[6].

3.5.1 Expray® Sample Collection

Expray® is designed to detect NC and NG on surfaces rather than in bulk material. Both penetrating and nonpenetrating cracks and other penetrating features were tested using Expray®. Additionally, drains and drainage traps external to the buildings were tested for potential explosives using Expray®. Expray® samples were obtained by wiping the surface to be tested with a special filter. Any residual explosive was picked up on the filter, which was then exposed to a series of three aerosol sprays that develop specific colors on the filter if explosives are present. Expray® samples were taken at each crack location in the buildings and at areas that appeared to be clean to test the method's propensity to generate false negatives. Table 3 lists explosive compounds that can be identified using this method.

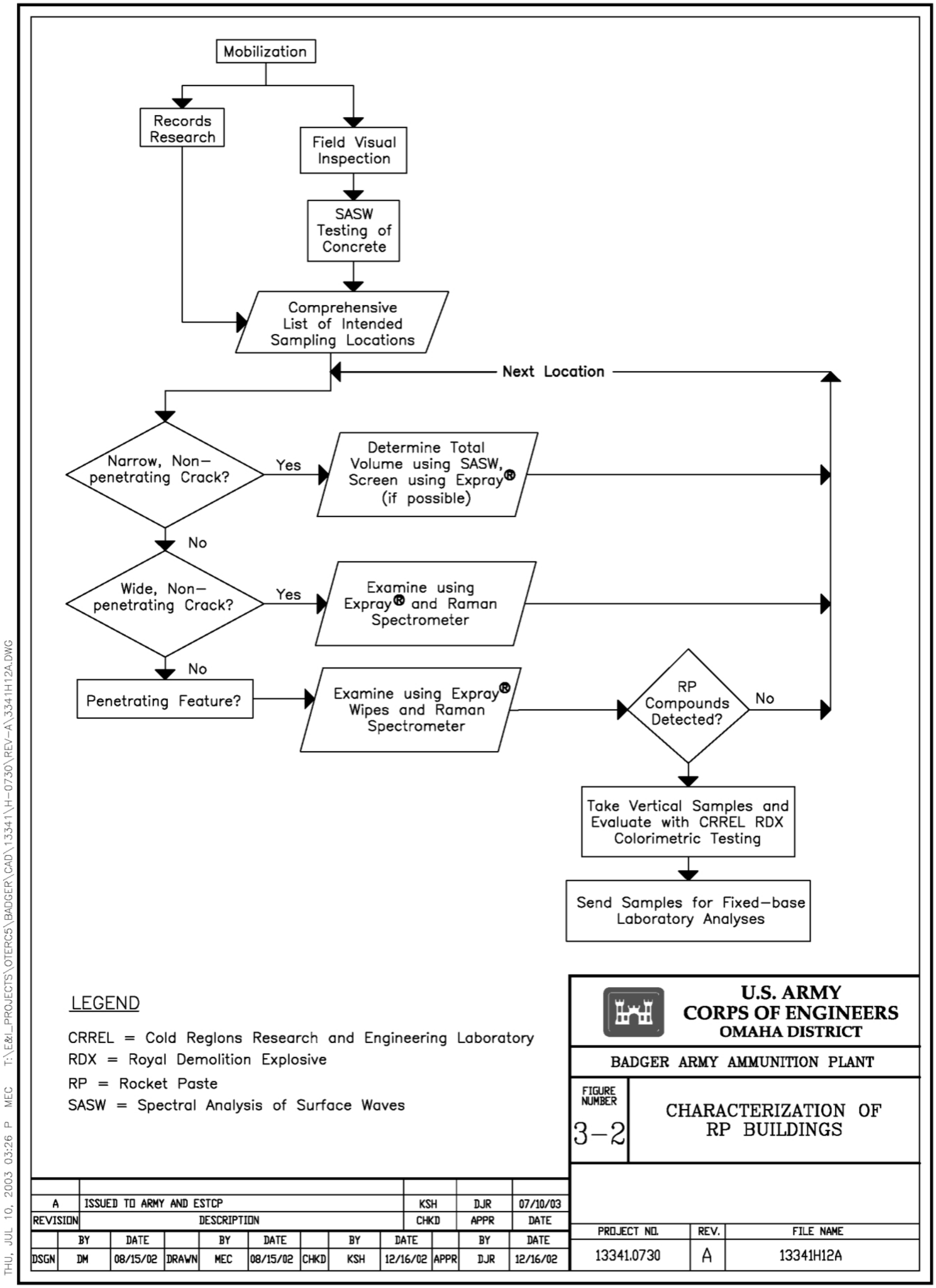

Figure 2. Characterization of RP Buildings.

Table 3. Target Compound List, Expray® Colorimetric Indicator Method.

EXPRAY - 1		EXPRAY - 2		EXPRAY - 3	
Polynitro-Aromatics		Nitrate-Esters Nitramines		Inorganic Nitrate Compounds	
Group A		Group B		Improvised	
Substance	**Color**	**Substance**	**Color**	**Substance**	**Color**
Ammonium picrate	Yellow	BTN	Pink	Ammonium nitrate	Pink
DDNP	Orange-brown	DEGN	Pink	Barium nitrate	Pink
DNT	Blue-green	EDDN	Pink	Black powder	Pink
Lead styphnate	Yellow	EGDN	Pink	Potassium nitrate	Pink
Nitroxylene	Brown	Haleite	Pink	Silver nitrate	Pink
Picric acid	Yellow	HMX	Pink	Sodium nitrate	Pink
Tetryl	Orange	NC	Pink	Strontium nitrate	Pink
TNB	Dark brown	NG	Pink		
TNT	Dark brown	Nitroguanidine	Pink		
Trinitro naphthalene	Violet	PETN	Pink		
		RDX	Pink		
		Semtex	Pink		
		Smokeless powder	Pink		
		Tetryl	Pink		

3.5.2 Raman Spectrometer Access and Sampling

Raman testing equipment was portable and easily moved between sampling locations. The equipment was powered using a 12-volt car battery and an appropriate converter. No other utilities were required for the operation of this equipment.

The Raman spectrometer was used to investigate features in the concrete that were large enough to admit the probe. Initially, it was used to determine if RP materials were present in cracks on the floor and foundation surfaces. After sampling locations for the underlying soils were chosen, materials that block the crack, joint, or penetrating feature were removed using nonsparking tools and/or high-pressure abrasive water jets.

Raman spectroscopy was used to evaluate the soil underlying the foundation once access had been provided. The Raman spectrometer was used to detect any particles of RP that may have accumulated in or on the soil underlying the penetrating feature. The probe was worked along the entire length of the bottom of each joint or crack to look for the presence of RP or other residue. Suspected particles were analyzed by the spectrometer.

3.5.3 Concrete Coring – Soil Sampling Access

Access to the soils at the bottom of a penetrating feature was accomplished using a water jet cutting system. The system was used to cut holes through the concrete at the sample location. The location of the hole was determined on a case-by-case basis, depending on the location of the penetration through the floor and the results of surface Raman spectroscopy. The size of the hole was made large enough to accommodate the Raman probe/sampling equipment.

There were several operational issues encountered with the water jet cutting tool. In general, water jet cutting was a very safe method to cut through concrete that may have the possible presence of explosives. However, the water jet cutting tool disturbed samples by blowing cutting

water through the concrete into the soil, had limited access to sampling locations, needed to be on a level surface, had maintenance problems related to hydraulics, and could not penetrate through rebar or large aggregate.

3.5.4 Soil Sample Collection and Management

Soil samples were obtained from the top 1 foot of soil beneath the building slabs and placed in clean, wide-mouth glass bottles or jars. The sample size was approximately 300 grams. Because of the large amount of water introduced by the water jet cutting system, it was not necessary to add more water to minimize the explosive safety hazards associated with sampling. The samples were logged with the appropriate building and feature identifiers. The samples were handled as described in the Comprehensive Field Sampling Plan[7]. Field duplicate samples were obtained at the rate specified in the Demonstration Plan[6]. Equipment rinsate blanks were not obtained because all sampling equipment used in the demonstration was disposable.

3.5.5 Spike Sample Collection and Management

Soil samples were spiked with known quantities of NC and NG to evaluate analyte recovery by the onsite laboratory. Spiked samples were prepared by adding a known quantity of NC or NG standards to a weighed, uncontaminated, and representative soil sample. A blank sample was also prepared and analyzed for each of the tests. Each spiked sample was also submitted to the offsite laboratory for analysis of NC or NG using the laboratory reference methods.

3.6 ANALYTICAL PROCEDURES

Field and laboratory analytical procedures detailed in Appendix D of the Demonstration Plan[6] were followed. Table 4 provides a summary of the analyses performed on samples.

Table 4. Summary of Sampling and Analyses Performed.

Parameter	Matrix	Analytical Method	No. Field Locations	No. Field Samples	No. Field Duplicates	Total No. Samples
Crack evaluation	Concrete	SASW	160	160	0	160
		Expray®	96	96	0	96
		Raman	88	88	0	88
NG/NC	Soil	Raman	35	35	0	35
		Expray®	31	31	0	31
		CRREL (SW-846 Method 8510)	35	50	4	54
		SW-846 method 8332 (NG)	35	41	7	48
		Army automated NC analysis	35	50	9	59
		EPA method 353.2 (Nitrogen, nitrate + nitrite)	15	15	4	19
SVOCs	Soil	SW-846 method 8270C	35	35	4	39
Lead and copper	Soil	SW-846 method 6020	35	35	4	39
Aluminum	Soil	SW-846 method 6010B	35	35	4	39

Notes:
SVOC = semivolatile organic compound

Field procedures used during the demonstration included Expray® wipes, Raman spectroscopy, and the CRREL RDX method. Samples from these field procedures were compared against laboratory reference methods, which included the following procedures:

- NG—SW-846 method 8332.
- SVOCs—SW-846 method 8270C.
- NC—U.S. Army automated nitrocellulose analysis.
- Metals—Lead and copper using SW-846 method 6020; aluminum using SW-846 method.
- Nitrates and nitrites—EPA method 353.2.

Additionally, spiked samples were prepared to test the CRREL RDX method against known quantities of NG and NC. Six samples were spiked with NG and analyzed using CRREL RDX method and SW-846 method 8332. Six samples were also spiked with NC and were analyzed using the CRREL RDX method and the Army automated nitrocellulose analysis.

4.0 PERFORMANCE ASSESSMENT

4.1 PERFORMANCE DATA

4.1.1 Field and Laboratory Results

The principal goal of the project was to compare the Raman spectroscopy, Expray® colorimetric, and CRREL RDX method field testing results with the off-site, fixed-base laboratory analytical results. Paired results between the field work and off-site laboratory were compared for the presence or absence of a particular compound and analytical value. Because results of the demonstration did not provide data that could be compared with a rigorous statistical program, calculations of relative percent difference (RPD) and absolute difference were not performed.

Demonstration results for each of the five buildings that were evaluated are discussed in depth in the Phase I Final Report, Rocket Paste Production Buildings Investigation[8]. For convenience, Table 5 provides a side-by-side comparison of the Raman, Expray®, CRREL RDX method, and offsite laboratory analyses for NC and NG. The samples with positive results are highlighted.

Table 5. Data Comparison — NC and NG Analyses.

Sample Location	Subsurface Raman	Expray® (2)	CRREL Method for NC/NG (mg/kg)	Army Automated NC Analysis (mg/kg)	Method 8332 for NG (mg/kg)
HH1C-1	ND	A	0.023 J	2.4 JS73	6.7 U
HH1C-1 (1)	NT	NT	ND	1.8 JT8	NT
HH2A-1	ND	A	ND	3.4 JS73	6.7 U
HH2D-1	ND	A, B	ND	6.8 JS73	6.7 U
HH4A-1	ND	ND	ND	4.6 JS73	6.7 U
HH4B-2	ND	A	0.8 J	3.9 JS73	6.7 U
HH4B-2 (1)	NT	NT	ND	1.7 JT8	NT
HH4D-1	ND	A	58.5	2.5 JS73	6.7 U
HH4D-1 (1)	NT	NT	ND	1.6 JT8	NT
HH7B-1	ND	ND	ND	2.7 JS73	6.7 U
BB1D-1	ND	B	0.23 J	21 JS73	6.7 U
BB1D-1 (1)	NT	NT	0.13 J	2.0 JT8	6.7 U
BB2B-1	Nitrocellulose	B	ND	8.5 JS73	6.7 U
BB2B-1 (1)	NT	NT	ND	1.9 JT8	NT
BB3B/3C-2	ND	B	ND	12 JS73	6.7 U
BB3B/3C-2 (1)	NT	NT	0.13 J	2.9 JT8	6.7 UJT8
BB3D-1	ND	B, C	ND	12 JS73	6.7 U
BB3D-1 (1)	NT	NT	0.03 J	1.5 JT8	6.7 UJT8
BB4B-1	ND	B	ND	16 JS73	6.7 U
BB5A-1	ND	B	0.07 J	3.1 JS73	6.7 U
BB5A-1 (1)	NT	NT	NT	2.2 JT8	NT
BB5C-1	ND	ND	8.9	8.3 JS73	6.7 U
BB5C-1 (1)	NT	NT	ND	4.1 JT8	6.7 UJT8
BT1A-5	ND	B	ND	2.3 JS73	6.7 U
BT1E-1	ND	A, B	ND	4.9 JS73	6.7 U
BT2C-1	ND	A, B	ND	2.4 JS73	6.7 U
BT2D-1	ND	ND	ND	11 JS73	6.7 U
BT2D-1 (1)	NT	NT	ND	1.9 JT8	NT
BL2A-1	ND	B	1.7	2.5 JS73	6.7 U
BL2A-1 (1)	NT	NT	ND	1.7 JT8	NT

Table 5. Data Comparison — NC and NG Analyses (continued)**.**

Sample Location	Subsurface Raman	Expray® (2)	CRREL Method for NC/NG (mg/kg)	Army Automated NC Analysis (mg/kg)	Method 8332 for NG (mg/kg)
BL4A-1	ND	B	ND	4.9 JS73	6.7 U
BL4B-1	ND	B	ND	4.7 JS73	6.7 U
BL5C-1	ND	NT	ND	3.1 JS73	6.7 U
BL6A-1	ND	B	ND	2.1 JS73	6.7 U
BL6B-1	ND	B	ND	9.8 JS73	6.7 U
BL6B-1 (1)	NT	NT	ND	1.8 JT8	NT
BL8A-1	ND	B	ND	2.8 JS73	6.7 U
BL9B-1	ND	NT	ND	3.7 JS73	6.7 U
RH1D-1	ND	B	ND	5.8 JS73	6.7 U
RH2B-2	ND	B	ND	5.2 JS73	6.7 U
RH3C-1	ND	ND	0.2 J	5.4 JS73	6.7 U
RH3C-1 (1)	NT	NT	ND	1.8 JT8	NT
RH6B-1	Nitrate	B	ND	2.6 JS73	6.7 U
RH6C-1	ND	B	ND	6.0 JS73	6.7 U
RH6F-2	ND	B	0.1 J	6.9 JS73	6.7 U
RH6F-2 (1)	NT	NT	0.27 J	2.4 JT8	6.7 UJT8
RH8E-1	ND	ND	0.1 J	3.3 JS73	6.7 U
RH8E-1 (1)	NT	NT	0.07 J	1.4 JT8	6.7 UJT8
RH12F-2	Nitrate	NT	ND	3.2 JS73	6.7 U
RH14H-2	ND	NT	ND	5.9 JS73	6.7 U

Notes:
(1) Additional sample taken on 8/27/02 – 8/28/02.
(2) Refer to Table 3 for compounds in each Expray® group.
ND = Nondetect
NT = Not taken
J = Estimated value. Compound was positively identified above method detection limit, but below reporting limit.
JS73 = Estimated value. Matrix spike recovery was outside control limits of 75 to 125% recovery at 73% recovery.
JT8 = Sample received at 8.5°C. False undetected values or low bias could be associated with the data as a result of degradation.
U = Compound not detected above the method detection limit of 6.7 mg/kg.

Figure 3 shows the relationship between the data obtained using each method. The information provided shows that little or no correlation exists between the field methods and the analytical laboratory results.

4.1.2 Spiked Samples Results

The field and laboratory analytical results for the five spiked NG samples are provided in Table 6. Correlation was noted between spiked concentrations, CRREL RDX method concentrations (field), and method 8332 concentrations (laboratory).

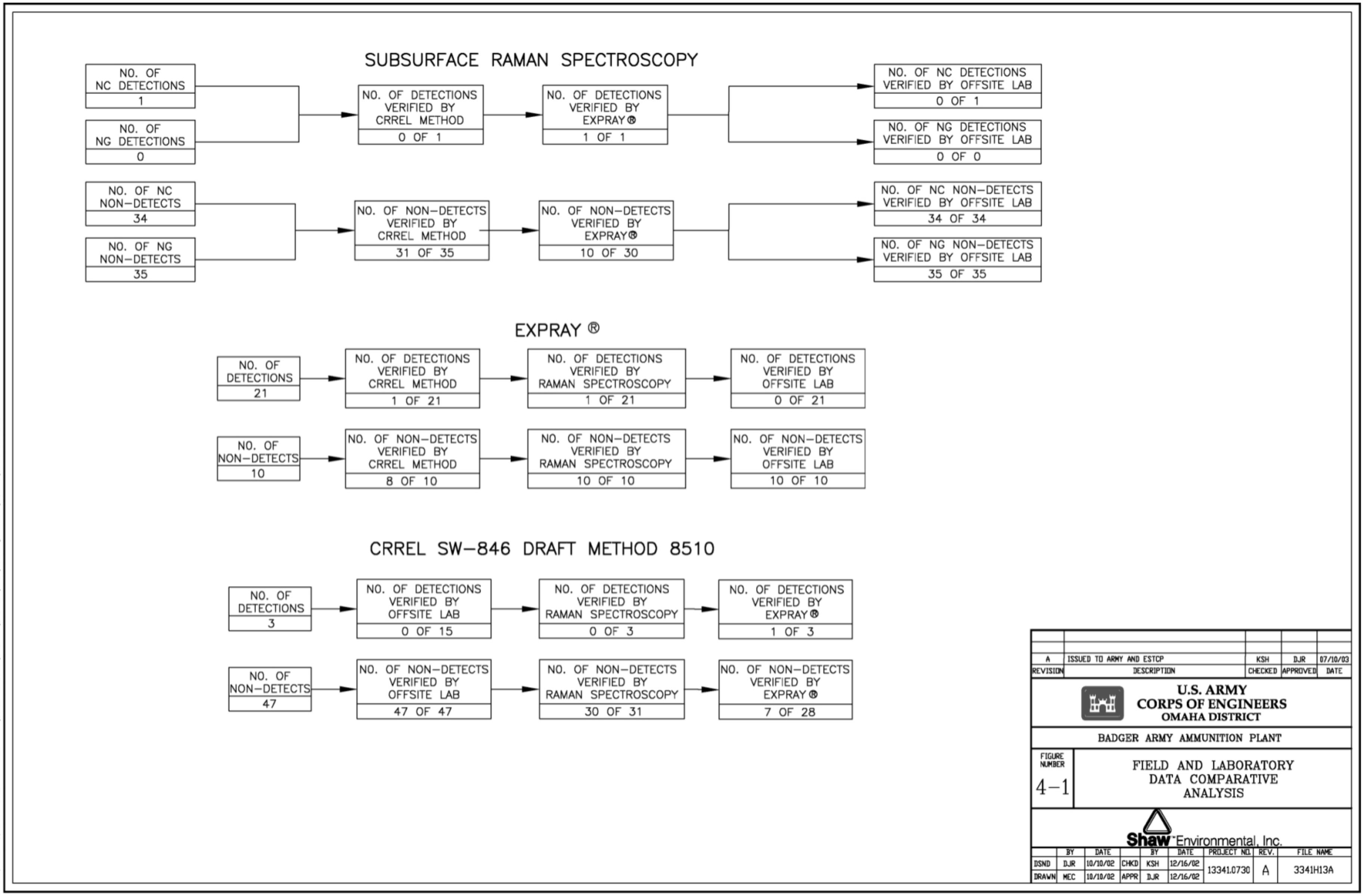

Figure 3. Field and Laboratory Data Comparative Analysis.

Table 6. NG Concentrations in Spiked Samples, Comparison of Field and Laboratory Methods.

Sample ID	NG Spiked Concentration (mg/kg)	CRREL RDX Method (mg/kg)	SW-846 Method 8332 (mg/kg)
A	15	16.0	30 JT22
B	10	10.1	12 JT22
C	6	5.6	7.7 JT22
D	3	3.1	3.3 JT22 (6.8)
E	1	1.4	6.8 UJT22
Blank	0	0.0	6.8 UJT22

Notes:
U = Compound not detected at reporting limit indicated in cell.
Have been qualified as T22 for a possible low bias as a result of degradation.

For the NG spiked sample results, a plot of the CRREL RDX method results, the SW-846 method 8332 results, and the spiked concentrations is shown in Figure 4.

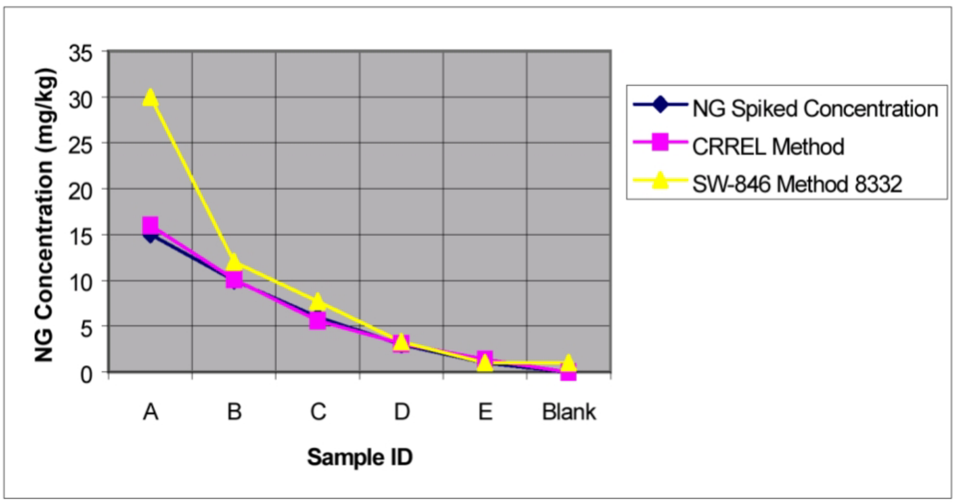

Figure 4. NG Concentrations in Spiked Soil Samples.

The field and laboratory analytical results for the five spiked NC samples are provided in Table 7. Preparation of the calibration curve for NC yielded absorbances that were comparable in all of the standards but did not correlate with the standard concentrations. Because of this, a calibration curve could not be produced, and soil concentrations of NC could not be quantified by the CRREL RDX method. Additionally, measurements of absorbances of the spiked samples yielded results that did not correlate with spiked concentrations. Measurement of NC standards, spike samples, CRREL RDX method (field), and the Army automated nitrocellulose analysis (laboratory) analytical results are shown in Table 7.

Table 7. NC Concentrations in Spiked Samples, Comparison of Field and Laboratory Methods.

Sample ID	NC Spiked Concentration (mg/kg)	CRREL NC Standard Absorbance	CRREL NC Spiked Sample Absorbance	CRREL RDX Method Absorbance	Army Automated Nitrocellulose Analysis (mg/kg)
A	400	0.154	NT	NM	2.3 UJT21
B	375	NT	0.125	NM	2.3 UJT21
C	200	0.153	0.154	NM	2.3 UJT21
D	100	0.120	0.036	NM	0.81 JT21 (2.3)
E	50	0.142	0.033	NM	3.0 JT21
Blank	0	0.022	0.032	NM	1.3 JT21 (2.0)

Notes:
NM = Not measured because of calibration curve failure
NT = Not taken
U = Compound not detected at reporting limit indicated in cell
JT21 = Estimated value. Reporting limit is shown in parentheses. Samples were received at 21°C. Sample results have been qualified as T21 for a possible low bias as a result of degradation.

For the NC spiked sample results, a plot of the Army automated nitrocellulose analysis results and the spiked concentrations is shown in Figure 5.

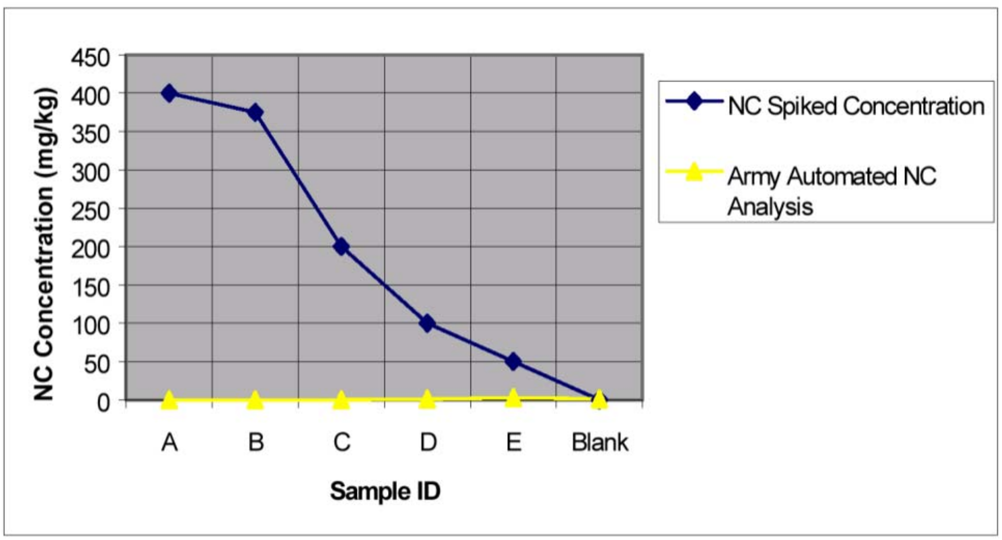

Figure 5. NC Concentrations in Spiked Soil Samples.

It is interesting to note that the laboratory reference method also failed to measure NC accurately. The Army automated nitrocellulose analysis was not designated for validation in this demonstration. It was assumed that this method would produce valid results for the entire range of concentrations that would be encountered. Originally, this method was used to measure NC in water and was intended for use in measurement of water pollution by NC fines. This method was modified for use with soils by adding additional steps designed to remove the NC from the soil matrix. Unfortunately, the method does not appear to be able to measure NC in soil, and no other method is known at this time that does measure NC in soils. Different possibilities were evaluated for the inconsistency of spiked soil NC results by the reference method, including incorrect reference standard, incorrect preparation of spiked samples, and inability of the laboratory reference method to measure NC accurately. During the spike sample study, an NC standard, Collodion U.S.P., was

used to spike the soil samples. Collodion is a liquid solution of NC that was added to each spike soil sample to achieve the concentrations shown in Table 7. One possible explanation for the results shown in the study is that during the initial washing step of the Army automated nitrocellulose analysis, the liquid NC was washed away along with the nitrates and nitrites. Since the laboratory reference method could not measure NC, it is not possible at this time to validate the technologies in the demonstration.

4.2 PERFORMANCE CRITERIA

The effectiveness of the demonstration was evaluated by confirming the performance of each of the technologies. Performance confirmation was based on field identification and analysis and fixed-base laboratory analytical results. Results of NC and/or NG identification tests were compared with on-site and off-site laboratory analytical results. Samples were also obtained to test for the presence of false negative identifications.

System performance was measured in definitive ways to the extent practical for both primary and secondary criteria, as initially identified in the Demonstration Plan[6]. This evaluation is presented in Table 8.

4.3 DATA ASSESSMENT

While the lack of significant quantities of energetic materials encountered on the floors and under the RP buildings is good news for the Army, it made the validation of the proposed methods difficult. Additionally, it appears that one of the laboratory reference methods, Army Automated Nitrocellulose Analysis, is unable to quantify NC accurately in soils.

In terms of the effectiveness of the technologies, some potential agreement between methods may have been observed in the demonstration; however, further validation of the technologies is required to prove this. Since the reference method for NC does not appear to measure NC accurately, any comparison must be qualified by the lack of bona fide data for NC. Tempered by this fact, comparison between Raman spectroscopy, CRREL RDX method, and EPA method 8332 generally indicate similar, low-level, qualified or nondetect results with the exception of two detections by the CRREL RDX method. Also, observations made in the field noted the absence of NC fibers in nearly all cases. Assuming that NC was not present in significant quantities, the methods do appear to agree that explosives were not present in significant quantities in the buildings investigated. Significant quantities are defined as quantities that would typically require evaluation from a safety perspective, which is the driver of this investigation. In any case, further validation should be done for all methods to ensure that the methods completely perform within performance metrics with valid reference method confirmation.

4.4 TECHNOLOGY COMPARISON

Inadequate information was obtained to make a technical comparison between in situ Raman spectroscopy testing, Expray®, and other technologies.

Based on limited data, the CRREL RDX method analysis for NG-spiked samples reveals greater sensitivity for NG quantification in comparison to the SW-846 method 8332.

Table 8. Performance Evaluation Against Primary and Secondary Criteria.

Performance Criteria	Expected Performance Metric (Pre-Demo)	Performance Confirmation Method	Actual Performance (Post-Demo)
Primary Criteria			
Compound identification	• NMT 5% false negative • NMT 10% false negative	Confirm by laboratory analysis. Data are evaluated on agreement of detection.	• Raman field method false negatives: 34 of 34 verified by lab • Expray® field method false negatives: 10 of 10 • CRREL field method false negatives: 47 of 47 • Raman field method false positives: 0 of 1 • Expray® field method false positives: 0 of 21 • CRREL field method false positives: 0 of 15 Note: Rate of false positives and false negatives may be incorrect because of possible interference/miscalculation in reference laboratory method (Army automated nitrocellulose analysis).
Compound concentration	• RPD NMT 20% • Correlation coefficient ≥0.95	Confirm by laboratory analysis. Data are evaluated on agreement of detection and concentration.	• CRREL method RPD = Not calculated • Correlation coefficient = Not calculated
Reliability	• Achieve identification and quantitation requirements in multiple locations/conditions	Confirm by laboratory analysis	• Not confirmed because of a lack of significant quantities of energetic material encountered
Ease of use	• Reduced crew size • Level of technical training • Need for special assistance or training during project • Operating crew can perform calibration and maintenance	Experienced from demonstration	• CRREL field method is easy to use with little specialized training and equipment. • Expray® field method is easy to use with little specialized training and equipment. • SASW operators require specialized training. Two operators are needed for efficient operation. • Raman operators require specialized training. • Trained SASW and Raman operators can perform calibration and maintenance on site.
Maintenance	• Percentage downtime when operations are scheduled • Routine maintenance required • Specialized personnel or equipment for maintenance activities	Experienced from demonstration	• CRREL field method did not use equipment that is subject to maintenance and repair. • Expray® field method did not use equipment that is subject to maintenance and repair. • SASW equipment downtime = 5 to 10% • Raman equipment downtime = 5 to 10% • High-pressure water jet cutting system required constant maintenance and repair: downtime = 20 to 25%

Table 8. Performance Evaluation Against Primary and Secondary Criteria (continued).

Performance Criteria	Expected Performance Metric (Pre-Demo)	Performance Confirmation Method	Actual Performance (Post-Demo)
Secondary Criteria			
Versatility	• Use conditions and ease of use under a variety of site conditions	Experience from demonstration	• Because of portability and size of equipment, SASW testing made it easy to transport. Interference at locations with multiple cracks and near sidewalls affected the usability of the SASW data at these locations. • CRREL field method can be used under a wide variety of site conditions. • Expray® field method can be used under a wide variety of site conditions. • Raman equipment is light and easy to transfer. • Raman probe must be held very still for measurement—requires tripod that affects the locations that can be sampled. • Raman probe size (1-inch) limits locations where Raman readings can be obtained. • High-pressure water jet cutting system needed a stable, flat base to operate—unable to cut concrete in ideal locations for sampling because of limitations of the cutting equipment.
Hazardous materials	• Volume of hazardous materials generated by project operations • Number of waste streams requiring characterization and disposal	Experience from demonstration	• Minimal hazardous materials were generated during project • Four ounces of spent acetone used in CRREL field method was containerized and disposed as hazardous waste.
Process waste	• Amount of investigation-derived waste generated by the project	Experience from demonstration	• Approximately 1,000 gallons of water containing soil brought to the surface was collected and treated at the BAAP pump and treat facility. Residual soil not vacuumed into the tank (about one hundred grams per location) was swept back into the sample location during cleanup activities.

Notes:
NMT = not more than
RPD = relative percent difference

5.0 COST ASSESSMENT

5.1 COST REPORTING

Project costs were tracked and are summarized in Table 9. The actual demonstration costs of Raman spectroscopy testing, Expray® testing, CRREL RDX method testing, SASW testing, and high-pressure water jet concrete cutting are provided in Table 10, Table 11, Table 12, Table 13, and Table 14. Shaw's planning and oversight costs are apportioned evenly between the five tasks on Table 10, Table 11, Table 12, Table 13, and Table 14. The unit cost per sample is provided for each activity.

The operational costs for each of the technologies are expected to be similar to those presented in Table 10, Table 11, Table 12, Table 13, and Table 14. For larger projects, the planning costs could be apportioned across a larger number of samples resulting in a slightly lower unit cost, but all other costs are expected to be similar.

5.2 COST COMPARISONS TO CONVENTIONAL AND OTHER TECHNOLOGIES

Table 15 provides the off-site analytical laboratory costs for the demonstration and includes the cost of obtaining the samples in the field. By comparing these costs with the costs of collecting and analyzing soil samples using the CRREL RDX method in Table 12, it is evident that considerable costs (35%) can be avoided using the field method as a substitute for and/or in addition to the off-site laboratory method. Since NG analysis by the CRREL RDX method was demonstrated to be a reliable field measurement tool, sites where NG analyses are needed could benefit from the use of this technology. For the other technologies and methods, additional demonstration should be performed to fully prove the technologies before any cost savings could be realized.

Table 9. Overall Demonstration Costs.

Cost Category	Sub Category	Details	Subcontractor Costs						Total Actual Demonstration Costs (Includes Shaw Costs)
			Concrete Cutting	SASW Testing	Analytical Laboratory	CRREL Testing	Raman Testing	Expray® Testing	
Start-up costs	Site characterization	N/A							
	Mobilization	Project planning costs		$2,692.88		$384.79	$1,252.45	$626.16	$19,956.28
		Coordination with client, regulatory agencies, and subcontractors		$2,657.91					$10,657.91
		Personnel travel to site	$1,272.96	$678.11		$238.00	$774.67	$387.29	$4,551.04
		Equipment travel to site	$3,000.00	$500.00					$3,500.00
		Shipping costs				$31.88	$103.75	$51.87	$529.25
Capital costs	Capital equipment purchase	N/A							
	Ancillary equipment purchase	N/A							
	Modifications	N/A							
	Structures installation	N/A							
	Engineering	N/A							
Operating costs	Capital equipment rental	SASW system		$3,000.00					$3,000.00
		Raman spectroscopy system					$5,000.00		$5,000.00
		High-pressure water jet cutting system	$20,115.68						$20,115.68
	Ancillary equipment rental	Pumps	$500.00						$500.00
		Vehicle(s)	$650.00			$85.00	$276.67	$138.32	$2,199.99
	Supervision	Salary	$8,413.40						$18,686.07
		Travel							$2,128.00
		Per diem	$850.00						$2,868.68
	Operator labor	Salary	$7,500.00	$13,149.01		$1,565.96	$5,097.08	$2,548.27	$29,860.32
		Travel – on-site		$336.86					$336.86
		Per diem	$1,275.00	$545.80		$144.50	$470.34	$235.14	$2,670.78
	Training	OSHA							
		Procedures							
	Maintenance	High-pressure hydraulic cutter							
		Other equipment (specified)							
	Consumables	Expray® kit(s)						$487.80	$487.80
		Personal protective equipment	$500.00						$500.00
		Laboratory supplies – CRREL method				$2,279.67			$2,279.67
		Fuel	$400.00	$32.59					$510.65
		Tools							$294.60
		Other (specified): TNT standards				$74.75			$74.75

Table 9. Overall Demonstration Costs (continued).

Cost Category	Sub Category	Details	Subcontractor Costs						Total Actual Demonstration Costs (Includes Shaw Costs)
			Concrete Cutting	SASW Testing	Analytical Laboratory	CRREL Testing	Raman Testing	Expray® Testing	
Operating costs (continued)	Residual waste handling	N/A							
	Off-site disposal	Hazardous waste (if any)							
	Analytical laboratory costs	NG			$5,336.00				$5,336.00
		NC			$4,366.00				$4,366.00
		Shipping costs			$142.56				$142.56
		Data validation							
	Long-term monitoring								
Indirect costs	Equipment repair	N/A							
Demobilization	Housekeeping	Other (specified)							
		Site cleanup/maintenance							
		Personnel travel from site	$1,272.96	$678.11		$238.00	$774.67	$387.29	$4,551.04
		Equipment travel from site	$3,000.00	$500.00					$3,500.00
		Shipping costs				$31.88	$103.75	$51.87	$318.77
		TOTAL	$48,750.00	$24,771.27	$9,844.56	$5,074.42	$13,853.37	$4,914.02	$148,922.68

Table 10. Demonstration Costs, Raman Spectroscopy Testing.

Cost Category	Sub Category	Details	Apportioned Shaw Costs	Raman Testing Subcontractor	Total Actual Demonstration Costs
Start-up costs	Site characterization	N/A			
	Mobilization	Project planning costs	$3,000.00	$1,252.45	$4,252.45
		Coordination with client, regulators, and subcontractors	$1,600.00		$1,600.00
		Personnel travel to site	$240.00	$774.67	$1,014.67
		Equipment travel to site			
		Shipping costs	$68.35	$103.75	$172.10
Capital costs	Capital equipment purchase	N/A			
	Ancillary equipment purchase	N/A			
	Modifications	N/A			
	Structures installation	N/A			
	Engineering	N/A			
Operating costs	Capital equipment rental	SASW system			
		Raman spectroscopy system		$5,000.00	$5,000.00
		High-pressure water jet cutting system			
	Ancillary equipment rental	Pumps			
		Vehicle(s)	$210.00	$276.67	$486.67
	Supervision	Salary	$2,054.53		$2,054.53
		Travel	$425.60		$425.60
		Per diem	$403.74		$403.74
	Operator labor	Salary		$5,097.08	$5,097.08
		Travel – on-site			
		Per diem		$470.34	$470.34
	Training	OSHA			
		Procedures			
	Maintenance	High-pressure water jet cutting system			
		Other equipment (specified)			
	Consumables	Expray® kit(s)			
		Personal protective equipment			
		Laboratory supplies - CRREL method			
		Fuel	$15.61		$15.61
		Tools	$58.92		$58.92
		Other (specified): TNT standard			
	Residual waste handling	N/A			
	Off-site disposal	Hazardous waste (if any)			
	Analytical laboratory costs	NG			
		NC			
		Shipping costs			
		Data validation			
	Long-term monitoring	N/A			
Indirect costs	Equipment repair	Other (specified)			
Demobilization	Housekeeping	Site cleanup/maintenance			
		Personnel travel from site	$240.00	$774.67	$1,014.67
		Equipment travel from site			
		Shipping costs	$26.25	$103.75	$130.00
		TOTAL	$8,343.01	$13,853.38	$22,196.38
				Number of sample locations	127
				Unit cost per sample	$174.77

Table 11. Demonstration Costs, Expray® Testing.

Cost Category	Sub Category	Details	Apportioned Shaw Costs	Expray® Testing Subcontractor	Total Actual Demonstration Costs
Start-up costs	Site characterization	N/A			
	Mobilization	Project planning costs	$3,000.00	$626.16	$3,626.16
		Coordination with client, regulators, and subcontractors	$1,600.00		$1,600.00
		Personnel travel to site	$240.00	$387.29	$627.29
		Equipment travel to site			
		Shipping costs	$68.35	$51.87	$120.22
Capital costs	Capital equipment purchase	N/A			
	Ancillary equipment purchase	N/A			
	Modifications	N/A			
	Structures installation	N/A			
	Engineering	N/A			
Operating costs	Capital equipment rental	SASW system			
		Raman spectroscopy system			
		High-pressure water jet cutting system			
	Ancillary equipment rental	Pumps			
		Vehicle(s)	$210.00	$138.32	$348.32
	Supervision	Salary	$2,054.53		$2,054.53
		Travel	$425.60		$425.60
		Per diem	$403.74		$403.74
	Operator labor	Salary		$2,548.27	$2,548.27
		Travel – on-site			
		Per diem		$235.14	$235.14
	Training	OSHA			
		Procedures			
	Maintenance	High-pressure water jet cutting system			
		Other equipment (specified)			
	Consumables	Expray® kit(s)		$487.80	$487.80
		Personal protective equipment			
		Laboratory supplies - CRREL method			
		Fuel	$15.61		$15.61
		Tools	$58.92		$58.92
		Other (specified): TNT standard			
	Residual Waste Handling	N/A			
	Off-site disposal	Hazardous waste (if any)			
	Analytical laboratory costs	NG			
		NC			
		Shipping costs			
		Data validation			
	Long-term monitoring	N/A			
Indirect costs	Equipment repair	Other (specified)			
Demobilization	Housekeeping	Site cleanup/maintenance			
		Personnel travel from site	$240.00	$387.29	$627.29
		Equipment travel from site			
		Shipping costs	$26.25	$51.87	$78.12
		TOTAL	$8,343.01	$4,914.02	$13,257.03
				Number of sample locations	123
				Unit cost per sample	$107.78

Table 12. Demonstration Costs, CRREL RDX Method.

Cost Category	Sub Category	Details	Apportioned Shaw Costs	CRREL Testing Subcontractor	Actual Demonstration Costs
Start-up costs	Site characterization	N/A			
	Mobilization	Project planning costs	$3,000.00	$384.79	$3,384.79
		Coordination with client, regulators, and subcontractors	$1,600.00		$1,600.00
		Personnel travel to site	$240.00	$238.00	$478.00
		Equipment travel to site			
		Shipping costs	$68.35	$31.88	$100.23
Capital costs	Capital equipment purchase	N/A			
	Ancillary equipment purchase	N/A			
	Modifications	N/A			
	Structures installation	N/A			
	Engineering	N/A			
Operating costs	Capital equipment rental	SASW system			
		Raman spectroscopy system			
		High-pressure water jet cutting system			
	Ancillary equipment rental	Pumps			
		Vehicle(s)	$210.00	$85.00	$295.00
	Supervision	Salary	$2,054.53		$2,054.53
		Travel	$425.60		$425.60
		Per diem	$403.74		$403.74
	Operator labor	Salary		$1,565.96	$1,565.96
		Travel – on-site			
		Per diem		$144.50	$144.50
	Training	OSHA			
		Procedures			
	Maintenance	High-pressure water jet cutting system			
		Other equipment (specified)			
	Consumables	Expray® kit(s)			
		Personal protective equipment			
		Laboratory supplies - CRREL method		$2,279.67	$2,279.67
		Fuel	$15.61		$15.61
		Tools	$58.92		$58.92
		Other (specified): TNT standard		$74.75	$74.75
	Residual waste handling	N/A			
	Off-site disposal	Hazardous waste (if any)			
	Analytical laboratory costs	NG			
		NC			
		Shipping costs			
		Data validation			
	Long-term monitoring	N/A			
Indirect costs	Equipment repair	Other (specified)			
Demobilization	Housekeeping	Site cleanup/maintenance			
		Personnel travel from site	$240.00	$238.00	$478.00
		Equipment travel from site			
		Shipping costs	$26.25	$31.88	$58.13
		TOTAL	$8,343.01	$5,074.42	$13,417.43
				Number of sample locations	35
				Unit cost per sample	$383.36

Table 13. Demonstration Costs, Spectral Analysis of Surface Waves Testing.

Cost Category	Sub Category	Details	Apportioned Shaw Costs	SASW Testing Subcontractor	Total Actual Demonstration Costs
Start-up costs	Site characterization	N/A			
	Mobilization	Project planning costs	$3,000.00	$2,692.88	$5,692.88
		Coordination with client, regulators, and subcontractors	$1,600.00	$2,657.91	$4,257.91
		Personnel travel to site	$240.00	$678.11	$918.11
		Equipment travel to site		$500.00	$500.00
		Shipping costs	$68.35		$68.35
Capital costs	Capital equipment purchase	N/A			
	Ancillary equipment purchase	N/A			
	Modifications	N/A			
	Structures installation	N/A			
	Engineering	N/A			
Operating costs	Capital equipment rental	SASW system		$3,000.00	$3,000.00
		Raman spectroscopy system			
		High-pressure water jet cutting system			
	Ancillary equipment rental	Pumps			
		Vehicle(s)	$210.00		$210.00
	Supervision	Salary	$2,054.53		$2,054.53
		Travel	$425.60		$425.60
		Per diem	$403.74		$403.74
	Operator labor	Salary		$13,149.01	$13,149.01
		Travel – on-site		$336.86	$336.86
		Per diem		$545.80	$545.80
	Training	OSHA			
		Procedures			
	Maintenance	High-pressure water jet cutting system			
		Other equipment (specified)			
	Consumables	Expray® kit(s)			
		Personal protective equipment			
		Laboratory supplies - CRREL method			
		Fuel	$15.61	$32.59	$48.20
		Tools	$58.92		$58.92
		Other (specified): TNT standard			
	Residual waste handling	N/A			
	Off-site disposal	Hazardous waste (if any)			
	Analytical laboratory costs	NG			
		NC			
		Shipping costs			
		Data validation			
	Long-term monitoring	N/A			
Indirect costs	Equipment repair	Other (specified)			
Demobilization	Housekeeping	Site cleanup/maintenance			
		Personnel travel from site	$240.00	$678.11	$918.11
		Equipment travel from site		$500.00	$500.00
		Shipping costs	$26.25		$26.25
		TOTAL	$8,343.01	$24,771.27	$33,114.28
				Number of sample locations	160
				Unit cost per sample	$206.96

Table 14. Demonstration Costs, High-Pressure Water Jet Cutting System.

Cost Category	Sub Category	Details	Apportioned Shaw Costs	Raman Testing Subcontractor	Total Actual Demonstration Costs
Start-up costs	Site characterization	N/A			
	Mobilization	Project planning costs	$3,000.00		$3,000.00
		Coordination with client, regulators, and subcontractors	$1,600.00		$1,600.00
		Personnel travel to site	$240.00	$1,272.96	$1,512.96
		Equipment travel to site		$3,000.00	$3,000.00
		Shipping costs	$68.35		$68.35
Capital costs	Capital equipment purchase	N/A			
	Ancillary equipment purchase	N/A			
	Modifications	N/A			
	Structures installation	N/A			
	Engineering	N/A			
Operating costs	Capital equipment rental	SASW system			
		Raman spectroscopy system			
		High-pressure water jet cutting system		$20,115.68	$20,115.68
	Ancillary equipment rental	Pumps		$500.00	$500.00
		Vehicle(s)	$210.00	$650.00	$860.00
	Supervision	Salary	$2,054.53	$8,413.40	$10.467.93
		Travel	$425.60		$425.60
		Per diem	$403.74	$850.00	$1,253.74
	Operator labor	Salary		$7,500.00	$7,500.00
		Travel – on-site			
		Per diem		$1,275.00	$1,275.00
	Training	OSHA			
		Procedures			
	Maintenance	High-pressure water jet cutting system			
		Other equipment (specified)			
	Consumables	Expray® kit(s)			
		Personal protective equipment		$500.00	$500.00
		Laboratory supplies - CRREL method			
		Fuel	$15.61	$400.00	$415.61
		Tools	$58.92		$58.92
		Other (specified): TNT standard			
	Residual waste handling	N/A			
	Off-site disposal	Hazardous waste (if any)			
	Analytical laboratory costs	NG			
		NC			
		Shipping costs			
		Data validation			
	Long-term monitoring	N/A			
Indirect costs	Equipment repair	Other (specified)			
Demobilization	Housekeeping	Site cleanup/maintenance			
		Personnel travel from site	$240.00	$1,272.96	$1,512.96
		Equipment travel from site		$3,000.00	$3,000.00
		Shipping costs	$26.25		$26.25
		TOTAL	$8,343.01	$48,750.00	$57,093.01
				Number of sample locations	35
				Unit cost per sample	$1,631.23

Table 15. Conventional Off-Site Analytical Laboratory Costs, NC/NG Analyses.

Cost Category	Sub Category	Details	Apportioned Shaw Costs	Off-Site Analytical Laboratory	Total Actual Demonstration Costs
Start-up costs	Site characterization	N/A			
	Mobilization	Project planning costs	$3,000.00		$3,000.00
		Coordination with client, regulators, and subcontractors	$1,600.00		$1,600.00
		Personnel travel to site	$240.00		$240.00
		Equipment travel to site			
		Shipping costs	$68.35		$68.35
Capital costs	Capital equipment purchase	N/A			
	Ancillary equipment purchase	N/A			
	Modifications	N/A			
	Structures installation	N/A			
	Engineering	N/A			
Operating costs	Capital equipment rental	SASW system			
		Raman spectroscopy system			
		High-pressure water jet cutting system			
	Ancillary equipment rental	Pumps			
		Vehicle(s)	$210.00		$210.00
	Supervision	Salary	$2,054.53		$2,054.53
		Travel	$425.60		$425.60
		Per diem	$403.74		$403.74
	Operator labor	Salary			
		Travel – on-site			
		Per diem			
	Training	OSHA			
		Procedures			
	Maintenance	High-pressure water jet cutting system			
		Other equipment (specified)			
	Consumables	Expray® kit(s)			
		Personal protective equipment			
		Laboratory supplies - CRREL method			
		Fuel	$15.61		$15.61
		Tools	$58.92		$58.92
		Other (specified): TNT standard			
	Residual waste handling	N/A			
	Off-site disposal	Hazardous waste (if any)			
	Analytical laboratory costs	NG		$5,336.00	$5,336.00
		NC		$4,366.00	$4,366.00
		Shipping costs		$142.56	$142.56
		Data validation			
	Long-term monitoring	N/A			
Indirect costs	Equipment repair	Other (specified)			
Demobilization	Housekeeping	Site cleanup/maintenance			
		Personnel travel from site	$240.00		$240.00
		Equipment travel from site			
		Shipping costs	$26.25		$26.25
		TOTAL	$8,343.01	$9,844.56	$18,187.57
				Number of sample locations	35
				Unit cost per sample	$519.64

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6.0 IMPLEMENTATION ISSUES

6.1 COST OBSERVATIONS

Costs were affected by safety concerns related to obtaining samples below slabs that may contain residuals of explosives in quantities that would present a hazard. Water jet cutting was chosen as a method to provide safe access to subslab samples. This method was expensive ($1,690/ sample) and caused disturbance to the sample itself. In the future, a different method of obtaining samples should be investigated. Use of a different concrete cutting method may affect costs, although conversations with vendors indicate that costs using alternative cutting methods, such as remote coring using a diamond bit cutter, may be similar to the costs shown in Table 9. In this way, disturbance to samples could be minimized. Costs related to concrete cutting will also be affected by the thickness and strength of the concrete slab.

In general, costs did not differ substantially from estimates. Additional samples were required to investigate quantification of NC. These samples would not be required during normal operations. Personnel used for Expray® application, Raman spectroscopy, and CRREL RDX method analyses were experienced in the use of these technologies, so a substantial change in cost related to learning curve effects is not anticipated.

6.2 PERFORMANCE OBSERVATIONS

It is difficult to comment on the performance of these technologies based on the results of the demonstrations and, in particular, the failure of the Army Automated Nitrocellulose Analysis. Some potential agreement between methods may have been observed in the demonstration. In any case, further validation should be done for all methods to show that the methods completely perform within performance metrics with valid reference method confirmation.

The Raman equipment requires concentrations of energetic material of at least 1% (10,000 mg/kg) in soils for detection. Since none of the soils encountered during the study contained concentrations close to that range, the study was unable to quantify levels of explosives that might be reliably detected using Raman spectroscopy.

Expray® analysis did detect the presence of Group A, B, and C compounds in various areas at BAAP. When compared to all of the other methods, it appears that there are potentially many more false positive results with Expray® than with other methods. Expray® has a variety of interferences, particularly with Group C compounds, which include inorganic nitrates. Any fertilizer containing nitrates will react to Expray®, and field experience has shown that adding too much of the sprays could cause a color change. In terms of Group A compounds, it is believed that an interference or interferences exist in the soil matrix, which may have caused a false positive detection.

The false negative rate for the Expray® analysis was 0% when compared with the laboratory reference methods (assuming that low-level, qualified results show lack of significant quantities of explosives). Overall, when looking at false positive rates, it appears that Expray® does not pass the performance metrics. However, when looking at false negative rates, Expray® does pass

the performance metrics. In certain situations, Expray® might still be used to indicate lack of explosive compounds, given its low rate of false negatives. Further validation should be done in all cases to ensure that methods completely perform within performance metrics with valid reference method confirmation.

The CRREL RDX method (the field method) yielded lower NG concentrations than SW-846 Method 8332 (the off-site laboratory method), but the field method results were actually closer to the known spiked concentrations than the laboratory results. It appears that the CRREL RDX method may more accurately predict the NG concentrations in soils than SW-846 Method 8332. Another advantage of the CRREL method is that it can detect NG at concentrations of 1 mg/kg in soils, whereas method 8332 offers a reporting limit of greater than 6 mg/kg. It also appears that the CRREL RDX method can detect NC in soils but cannot adequately measure NC concentrations.

6.3 SCALE-UP

Because of a lack of sufficient amount of explosive materials at the sampling locations, Raman technology was not proven as a method for determining the presence of explosives. However, Raman technology in general has been proven for detecting a variety of different compounds. Assuming that explosives are present in detectable (>1% explosives) quantities, Raman technology may be ready for implementation for investigations related to safety concerns. Further characterization with buildings containing higher levels of explosives is needed to characterize performance. For wider application of the technology, such as investigating inside pipes, new specialized probes need to be developed and demonstrated that include the ability to view the sample remotely at long distances. Since Raman spectroscopy requires energetic material concentrations greater than 10,000 mg/kg (>1%) for detection, the utility of this technology for regulatory compliance evaluations is limited.

The CRREL RDX method appears to be a good method for detection of and quantification of NG in soils. Because of the lack of significant quantities at the sampling locations, further characterization with buildings containing higher levels of explosives should be performed to characterize performance adequately. The CRREL RDX method also appears to be able to detect NC in soils. The method was not able to quantify NC in soils, possibly because of the mix of reagents that provide the color that was measured as part of the testing process. Further investigation into the proper mix of reagents may be needed to determine if the CRREL RDX method can be used to measure NC in soils.

Expray® did not appear to be an effective way of finding large amounts of explosives since it is fairly sensitive and appears to have a number of possible interferences that cause false positives. However, combined with SASW technology, in certain situations Expray® may still be useful for identifying areas that do not contain explosive contamination in buildings.

Following a successful validation of these technologies, the most effective pathway for transferring the technologies would be through the manufacturers themselves, in the case of Expray® and CRREL RDX method. Manufacturers of the test kits are actively exhibiting and marketing the kits, and additional validation of the procedures for measurement of NC and NG could be presented easily as part of the manufacturers' literature. Applied Research Associates,

the contractor used for the demonstration, is actively marketing the Raman spectroscopy technology. Through these channels, the technologies can move from demonstration-scale to full-scale implementation once successfully validated.

6.4 LESSONS LEARNED

6.4.1 Concrete Coring Methods

The most important lesson learned was the poor performance of the concrete cutting method that was used in this demonstration. For future efforts that require coring through concrete foundations to obtain samples in potentially explosive settings, it would be worthwhile to consider remotely operated wet concrete sawing techniques rather than the high-pressure cutting system. Additionally, an effort should be made to find equipment that can fit in tight or restricted spaces, as well as immediately next to walls, so that equipment access is not an issue.

6.4.2 Lack of Energetic Materials

It would be prudent to design technology demonstrations for sites with known levels of contamination that are sufficiently high to be detected by the demonstration technologies employed. A significant effort was expended searching for buildings at BAAP that possibly contained NC and NG, but it appears that additional time should have been spent on finding candidate buildings that contained a good range of energetic materials.

6.4.3 NC Analyses

Results of the spike sample study for NC indicate that the laboratory reference method, Army automated nitrocellulose analysis, failed to measure NC accurately in all cases. This reference method was used throughout the study as a measurement of NC, and its results were compared to the field method results. The failure of this reference method to measure NC accurately prevents validation of the field methods at this time. For future work, another method of measurement of NC in soils should be determined and proven effective. In particular, the method should ensure that NC alone could be isolated from other interferences that might exist in soil matrices. Currently, no other method of measurement of NC in soils is known.

6.4.4 Revised Sampling and Analysis Plan

To validate these technologies successfully in the near future, a revised sampling and analysis plan may be developed. The revised sampling and analysis plan should include information about each of the methods, as well as information to allow field personnel to isolate any interferences that may be encountered.

The revised sampling and analysis plan should include a different laboratory reference method for measurement of NC in soils. Additionally, access to soil samples should be specified so that a minimal disturbance to subslab soils occurs. This would ensure that potential explosives contamination that may be encountered on the surface of the soil is not moved or disturbed. Coring methods to allow for access to soil should also be specified to allow for as much access as possible. Where available, coring devices will be specified that can work in very confined

spaces, in drainways, and immediately next to walls. The coring device should also use as little water as possible to minimize disturbance to soil samples. For soil samples that are analyzed using the three field methods, extraction of the soil sample using acetone can be done, and the extract analyzed using the field methods, rather than the soil sample. In certain cases, this has proved successful in isolating NC and/or NG from interferences in the soil matrix. Unfortunately, this technique was learned following the field work. For the spiked soil tests, materials used in standards should be matched where possible with the types of materials found at the site. In particular, the NC standard used for spiking the soil samples was a liquid standard. NC found at the site is in a fiber form, and there is a possibility that the liquid standard was washed away during the analytical procedure for the reference method.

6.5 APPROACH TO REGULATORY COMPLIANCE AND ACCEPTANCE

The WDNR (the lead regulatory agency at BAAP) approved the Demonstration Plan[6] and has expressed considerable interest in characterizing the buildings at BAAP so that they may be safely transferred out of DoD control. Many other regulatory agencies find themselves in the position of desiring defendable characterization for DoD buildings, so that land transfer decisions can be made safely and effectively. The lack of significant quantities of energetic material found during the demonstration make acceptance of the technologies by the WDNR or other agencies unlikely because of lack of information. Further testing is necessary to gain regulatory acceptance of the approaches used.

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APPENDIX A

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